PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

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PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing *(day/month/year)*	08.11.2004
Applicant's or agent's file reference: X	**IMPORTANT NOTIFICATION**

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/GB 03/03031	14.07.2003	13.07.2002

Applicant
CHANCE & HUNT LIMITED et al

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

The applicant's attention is drawn to Article 33(5), which provides that the criteria of novelty, inventive step and industrial applicability described in Article 33(2) to (4) merely serve the purposes of international preliminary examination and that "any Contracting State may apply additional or different criteria for the purposes of deciding whether, in that State, the claimed inventions is patentable or not" (see also Article 27(5)). Such additional criteria may relate, for example, to exemptions from patentability, requirements for enabling disclosure, clarity and support for the claims.

Name and mailing address of the international preliminary examining authority:	Authorized Officer
European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Georgakopoulou, P Tel. +49 89 2399-6037

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference x	**FOR FURTHER ACTION**	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/GB 03/03031	International filing date *(day/month/year)* 14.07.2003	Priority date *(day/month/year)* 13.07.2002
International Patent Classification (IPC) or both national classification and IPC C08K3/34		
Applicant CHANCE & HUNT LIMITED et al		

1. This International preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 8 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 16 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the opinion
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
22.01.2004	08.11.2004
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized Officer Glomm, B Telephone No. +49 89 2399-7158

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB 03/03031

I. Basis of the report

1. With regard to the **elements** of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17))*:

Description, Pages

1-13 received on 27.10.2004 with letter of 27.10.2004

Claims, Numbers

1-22 received on 27.10.2004 with letter of 27.10.2004

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

   These elements were available or furnished to this Authority in the following language: , which is:

   ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).

   ☐ the language of publication of the international application (under Rule 48.3(b)).

   ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

   ☐ contained in the international application in written form.

   ☐ filed together with the international application in computer readable form.

   ☐ furnished subsequently to this Authority in written form.

   ☐ furnished subsequently to this Authority in computer readable form.

   ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

   ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

   ☐ the description, pages:

   ☐ the claims, Nos.:

   ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

   (Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	
	No:	Claims	1-22
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-22
Industrial applicability (IA)	Yes:	Claims	1-22
	No:	Claims	

2. Citations and explanations

see separate sheet

Cited documents:

D1: WO 98/08898 A (DU PONT DE NEMOURS AND COMPANY) 5 March 1998 (1998-03-05)
D2: EP-A-0 149 813 (BASF AG) 31 July 1985 (1985-07-31)
D3: EP-A-0 033 361 (HOECHST AG) 12 August 1981 (1981-08-12)
D4: EP-A-0 035 094 (HOECHST AG) 9 September 1981 (1981-09-09)
D5: US-A-5 643 980 (M. SHINDOH) 1 July 1997 (1997-07-01)
D6: GB-A-1 538 093 (CIBA-GEIGY AG) 10 January 1979 (1979-01-10)
D7: DE 28 00 891 A (MONTEDISON S.P.A.) 13 July 1978 (1978-07-13)
D8: GB-A-1 286 192 (FOKKER-VFW N.V.) 23 August 1972 (1972-08-23)

1. Amendments (Art. 34 (2) (b), second sentence PCT)

The newly filed set of claims 1 to 22, and also the newly filed description pages 1 to 13, clearly lack any supporting basis in the contents of the application papers as originally filed. Consequently, said amendment represents an undue extension of present application which violates the provisions of the Art. 34 (2) (b), second sentence PCT.

As regards the statement of the applicant on page 3, bottom paragraph of his letter dated 27.10.2004, the attention of the applicant is strongly drawn to the fact, that only the application papers as originally filed (i.e., as internationally filed) may constitute any basis for amendments in present case.

In the subsequent European regional stage, if any, the applicant is invited to file new claims and description, which both should be accurately based on the contents of the application papers as originally filed, while indicating clearly and expressly feature by feature the supporting basis in said original application

papers, in order to avoid immediate refusal of the application under the Arts. 97 (1) and 123 (2) EPC.

2. Novelty (Art. 33 (2) PCT)

In view of the above objection pointed out under section 1.), the novelty objection of previous communication is regarded as being still valid for the purposes of substantive preliminary international examination. Each of cited documents D1 to D8 discloses a composition of matter embodied therein a flame retardant material comprising a combination of a phosphorous containing material as specified in present independent main claim 1 (for relevant passages, see the corresponding International Search Report).

The attention of the applicant is drawn especially to the fact, that the parameter as specified in present (very broad and generally worded !) independent main claim 1 in lines 3/4 appears to be implicitly disclosed by each of said documents D1 to D8 in view of the principles of the established official rules of practice. Implicit disclosure corresponds to the fact, that the claimed product is regarded as being anticipated actually by said prior art documents, even if the claimed parameters as specified in the last two lines of present main claim 1 are not expressly mentioned therein, i.e., the parameters are regarded as being actually present in the prior art embodiments, but simply not determined and/or mentioned expressly.

Furthermore, the considerations in applicant's letter dated 27.10.2004 are not convincing at all for the following reasons 1 to 5:

1.) As regards the repeated discussion of process-related features and/or -advantages, the attention of the applicant is drawn to the fact, that present claim

1 is a (still very generally worded) product-claim, based on a "comprising"-wording, which does not exclude any further additives.

2.) Furthermore, for sake of completeness, in view of the established rules of practice, even the addition of process-related features to such a product-related claim may not render such claim novel, unless the product as such is not anticipated.

3.) Features appearing only in dependent claims (or optional features of independent claims) will never render any claim novel.

4.) Discussion of any specific advantages and/or unexpected effects of the claimed subject matter as repeatedly done in appplicant's said letter dated 27.10.2004 is a question of inventiveness only, and may also never render any claims novel.

5.) The disclosure of a prior art document is not to be limited unduly to the examples or preferred embodiments. Actually, the viewpoint of an average person skilled in the art when reading the whole document in its entirety is decisive.

To sum up, the applicant fails to discuss novelty of the claimed subject matter in view of the claimed combination of features of present main claim 1. Actually, the preliminary international examination authority still feels unable to identify any technical feature of present claim 1, which is not fully anticipated by each of said prior art documents D1 to D8.

Consequently, each of said documents D1 to D8 anticipates the subject matter of present claim 1.

The same considerations also relate to the additional features of the following claims 2 to 22 when taking into account the full disclosure of each of said

documents D1 to D8.

Therefore the subject matter of present application is not new in view of the disclosure of each of said documents D1 to D8.

3. Inventive Step (Art. 33 (3) PCT)

Providing an amended main claim which meets the requirements of Art. 33 (2) PCT, the applicant should relate the distinguishing feature to a surprising (unexpected) technical effect or make credible or plausible that the distinguishing feature is not derivable from the prior art teaching (Art. 33 (3) PCT).

4. Miscellaneous

The obscure parameter as specified in lines 3/4 of present main claim 1 appears to attempt a definition of the subject matter to be protected by means of the corresponding results to be achieved, rather than by means of clear and unambiguous technical features, such violating the Art. 6 PCT. The applicant therefore is invited to replace said objected terms by clear and unambiguous technical features based on suitable subclaims or relevant passages taken from the present description.

In order to improve the understanding and legibility of the application, in the subsequent European regional stage, if any, the documents D1 to D8 should be identified in the description additionally and the relevant background art disclosed

therein should be briefly discussed.

When filing amendments, any undue extension of the scope of the application should be avoided.

FLAME RETARDANT PRODUCTS

This invention relates to flame retardant products.

Flame retardant products exhibiting intumescent properties are well known. Such flame retardant products are incorporated in many compositions of matter, (host materials), especially thermoplastic polymer compositions. One such flame retardant product is a blend of a phosphoric acid producing catalyst, a charring agent and a blowing agent. The catalyst is a compound, e.g. ammonium polyphosphate, which when exposed to flame yields phosphoric acid. The charring agent can be a polyhydric alcohol, e.g. pentaerythritol, which decomposes and reacts with phosphoric acid to form a carbonaceous char. The blowing agent, e.g. melamine, when exposed to flame produces a non-flammable gas (e.g. N_2) which serves to foam and expand the carbonaceous char.

The above mentioned three component flame retardant products are powder additives which have processing limitations as they do not blend well with many compositions of matter (host materials), e.g. thermoplastics. In order to overcome these processing problems there have been attempts to encapsulate the flame retardant additives in inert polymers. However, there is a disadvantageous limit on the amount of such encapsulated flame retardant product that can be incorporated in the host materials and the encapsulants themselves are generally flammable materials.

Proprietary flame retardant products have appeared on the market which typically are reaction products of pentaerythritol and phosphate esters. These flame retardant products are melt blendable with host materials such as thermoplastic polymers. However, such proprietary flame retardant products have to be used in combination with other flame retardants. Furthermore, such proprietary materials do not contain blowing agents and so do not have the advantages of char foaming and expanding.

Conventional flame retardant compositions are described in the following prior art.

WO 98/08898 discloses thermosetting resin coatings, (e.g. melamine formaldehyde), for flame retardant compositions. These thermosetting coatings are there purely for their physical effect which is to lower the surface tension of the encapsulated flame retardant particles to that of host polymers.

EP 0149813A discloses a flame proofed thermoplastic moulding material (e.g. styrene polymers) which contains a three component flame retardant mixture of phenol formaldehyde resins; a nitrogen containing organic compound (e.g. urea) and a phosphorus containing organic compound. The phenol formaldehyde resin is a thermosetting resin which is included for its thermodynamic properties.

EP 0033361A describes the coating of the flame retardant ammonium phosphate with a condensation product of melamine and formaldehyde which is a thermosetting resin. The melamine formaldehyde resin serves to provide a waterproof coating for the ammonium phosphate. EP 0035094A makes a similar disclosure to EP 0033361 A.

US 5643980 discloses a flame retardant thermoplastic resin (e.g. a polyolefin) which contains a polyhydric alcohol-boric acid complex as a filler (e.g. a pentaerythritol complex) and the conventional flame retardant materials ammonium polyphosphate and melamine (or melamine derivatives).

GB 1538093 relates to aqueous compositions and intumescent foams (e.g. polyurethane forms) made therefrom comprising a urea formaldehyde resin as filler, phosphorous acid as a hardener and a carbonate of an aliphatic, araliphatic, heterocyclic-aliphatic or heterocyclic amine as a blowing agent. The urea formaldehyde resin is a thermosetting resin.

DE 2800891A discloses a self-extinguishing polymeric composition which contains as a flame retardant additive, a mixture of an ammonium or amine phosphate, a polyamide and a urea formaldehyde thermosetting resin. The polyamide is essential for flame retardant activity.

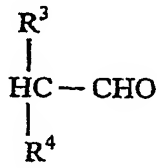
GB 1286192A discloses a thermosetting polymeric composition having conventional intumescent ingredients.

An object of the present invention is to provide a unique flame retardant product which overcomes the problems of known flame retardant products in that it is more readily blendable with many compositions of matter (host materials), particularly thermoplastic compositions, and thus imparts a higher degree of flame retardancy to the host materials.

According to the present invention, there is provided a flame retardant composition which comprises a mixture of a phosphorus containing compound which decomposes to produce phosphoric acid when exposed to flame and an oxygenated heterocyclic thermoplastic resin which is prepared by reacting an urea of the general formula (I)

(I)

where X is oxygen or sulphur and R^1 and R^2 are hydrogen, identical or different alkyl of 1 to 18 carbon atoms, aryl of 6 to 9 carbon atoms or aralkyl of 7 to 9 carbon atoms or may be an alkyleneurea radical, where alkylene is of 1 to 9 carbon atoms, with at least 2 moles of a CH-acidic aldehyde of the formula (II)



(II)

where R^3 is hydrogen and R^4 is alkyl, aryl or aralkyl, or R^3 and R^4 are identical or different alkyl, aryl or aralkyl, in the presence of a strong acid, to give a condensation product, and thereafter treating the product with an alkali metal alcoholate in an anhydrous medium.

Preferably, R^3 and R^4 in the compound of general formula (II) is alkyl of 1 to 10 carbon atoms, aryl of 6 to 9 carbon atoms or aralkyl of 7 to 9 carbon atoms.

Further preferably, the compound of general formula (I) is urea and the compound of general formula (II) is isobutyroaldehyde.

The phosphorus containing material preferably is selected from ammonium polyphosphate, sodium polyphosphate, potassium polyphosphate, melamine polyphosphate, melamine phosphate or mixtures thereof.

Advantageously, the phosphorus containing material is a mixture of ammonium polyphosphate and melamine phosphate.

The flame retardant composition may contain a blowing agent, suitably melamine or urea.

Preferably, the phosphorus containing material is encapsulated in the oxygenated heterocyclic thermoplastic resin.

Further preferably, the blowing agent and other ingredients of the flame retardant material are encapsulated in the oxygenated heterocyclic thermoplastic resin.

In this specification, the oxygenated heterocyclic thermoplastic resin is a resin prepared according to the process described in US Patent 4220751 (BASF) wherein the resin is a condensation product of an urea and a CH-acidic aldehyde. CH-acidic aldehydes are those where the carbon adjacent to the carbonyl group carries one or two hydrogen atoms.

In a preferred embodiment of the invention, the flame retardant composition comprises 25 to 60% by weight oxygenated heterocyclic thermoplastic resin; 0 to 75% by weight ammonium polyphosphate; 0 to 75% by weight melamine phosphate; and 0 to 45% by weight melamine with the proviso that ammonium polyphosphate or melamine phosphate essentially is present.

From another aspect the present invention is a composition of matter containing the flame retardant composition described and claimed herein.

The composition of matter may contain an amount of 5 to 90% by weight, preferably 10 to 45% by weight of the flame retardant composition. Higher inclusions may be desirable for masterbatches and systems requiring higher intumescent functionality.

Suitable compositions of matter (host materials), include thermoplastic polymers, thermosetting polymers, paper, reconstituted wood products and solvented systems (i.e. where the flame retardant material is dissolved in a solvent or mixtures of solvents).

Preferred host materials are polyolefins, particularly polypropylene.

From yet another aspect, the present invention is an article made from the composition of matter described above. Such articles can be made by compression moulding or injection moulding.

From yet another aspect, the present invention is a method of improving the flame retardant capability of a composition of matter by embodying in the composition of matter a flame retardant composition as described above.

Compositions of matter containing the flame retardant composition of the invention can be used in the manufacture of a wide variety of products and components for use in the electronic, construction and transport industries and can be incorporated into many structures including fire doors, vehicle passenger compartments, aircraft passenger and cargo areas as well as cargo storage containers and aircraft galley equipment, railway and underground carriages, cable trays (to prevent both loss of signal through the cable and passage of fire and heat along the cable tray itself), marine bulkheads, compressed gas and building structures.

Embodiments of the invention will now be described by way of example.

In the following examples of the invention the host material is polypropylene. The examples show that the Limiting Oxygen Index (LOI) of polypropylene incorporating the flame retardant material of the invention is increased. Since oxygen forms approximately 21% of normal atmosphere, thermoplastic polymers which have an LOI of 21% or less usually burn freely in air. If the inclusion of a material into the polymer increases the LOI of the polymer then this means that some degree of flame retardance is imparted to the polymer. As the LOI of the polymer increases above 21% then the polymer becomes increasingly difficult to ignite and also increasingly

likely to self extinguish. Generally speaking, once the LOI increases to above 30% then the polymer in effect is considered to be non-flammable and an LOI of 25% indicates good flame retardancy.

Successful polypropylene formulations containing a variety of examples of the flame retardant material of the invention are illustrated in the Tables below. All of the formulations contain the essential ingredients (a) the thermoplastic resin and (b) the phosphoric acid source (ammonium polyphosphate and/or melamine phosphate and some of the formulations also include melamine as a blowing agent).

The flame retardant material of the invention is not a simple combination of the powdered components but rather it is an extrudate. The ammonium polyphosphate, melamine phosphate and melamine are effectively encapsulated in the oxygenated heterocyclic thermoplastic resin during the extrusion process. This extrudate is normally produced as a chip (but with different equipment it could be made as a pellet or prill if required). The chip can be milled to a powder if this is considered desirable.

The finished flame retardant material has the appearance of a piece of dull white plastic. The product is virtually dust free and the chip size can be varied to suit end use requirements. The flame retardant material of the invention is a melt blendable product. The oxygenated heterocyclic thermoplastic resin casing is both part of the integral flame retardant mechanism but also makes the product melt blendable with many host materials. Compared to the traditional blends of flame retardants there is no pentaerythritol present. The oxygenated heterocyclic thermoplastic resin is the charring agent as well as giving the flame retardant material its melt blendable property.

It is to be noted that the flame retardant material of the invention is not a reaction product of its ingredients but rather is a physical blend of the ingredients. To our knowledge, no other non-halogen flame retardant uses this method of having an oxygenated heterocyclic thermoplastic resin incorporated which is part of the flame retardant system. Other flame retardant systems normally use inert polymers to either encapsulate the products or as an inert backbone onto which the flame retardant molecule is grafted.

In Table 1 below, the test samples are two and three component samples produced by compression and in Table 2, the test samples are four component samples produced by injection. Table 3 shows UL94 Vertical Burning Tests of polypropylene with various loadings of the four component sample of Example 9.

In the Tables, the following terms have the following meanings:

- PP means polypropylene
- APP means ammonium polyphosphate
- MP means melamine phosphate
- the "Level" column indicates the % w/w inclusion of the flame retardant product in untreated polypropylene.
- MFI refers to the Melt Flow Index – this gives an indication of how difficult the flame retardant addition makes the resulting polymer composition to process (in general the lower the MFI, the more difficult the polymer composition is to process). The MFI conditions were 230°C and a weight of 2.16kg.
- UL94 refers to a standard test of the Underwriters Laboratory.

The ammonium polyphosphate used in the examples was Exolit AP422 from Clariant. The oxygenated heterocyclic thermoplastic resin used was Laropal A81 which is an aldehyde resin obtained from BASF. The aldehyde resin, Laropal A101, again obtainable from BASF also could be used.

The melamine phosphate used was Melapur MP obtainable from Ciba Speciality Chemicals. The melamine phosphate provides both a phosphoric acid source for the char formation and a source of melamine and so provides dual function.

The quantities expressed in the Tables are weight percentages.

Table 1

Example	Aldehyde Resin	APP	MP	Melamine	Level	LOI
Blank PP	0	0	0	0	0	17
1	40	0	60	0	20	19
2	40	40	0	20	20	21
3	40	40	20	0	20	23
4	35	65	0	0	20	22.5
5	45	55	0	0	20	20

Table 2

Example	Aldehyde Resin	APP	MP	Melamine	Level	LOI	UL94 (1.6mm)
6	40	40	10	10	30	24.8	Full Burn
7	35	55	5	5	30	31.3	Full Burn
8	35	45	10	10	30	31.3	V0
9	35	50	7.5	7.5	30	33.2	V0

Table 3

Specimen Dimensions (mm):

Length: 125

Width: 13

Thickness: 0.8

Conditioning Procedure: 23°C and 50% relative humidity

Sample	UL94 Vertical Burning Test						
	Afterflame Time 1 (s)	Afterflame Time (s)	Total Time (s)	Burned to the clamp	Cotton ignited	Classification Single	Overall
20% Ex.9	1	0	1	N	Y	V-2	V-2
	0	0	0	N	Y	V-2	
	0	0	0	N	Y	V-2	
	0	0	0	N	Y	V-2	
	0	0	0	N	N	V-0	
	Total: 1		Av: 0				
25% Ex.9	0	0	0	N	Y	V-2	V-2
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	Total: 0		Av: 0				
25% Ex. 9 REPEAT	0	0	0	N	N	V-0	V-0
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	Total: 0		Av: 0				
30% Ex. 9	0	0	0	N	N	V-0	V-0
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	0	0	0	N	N	V-0	
	Total: 0		Av: 0				

Observations from the Tables

(i) It is possible to improve the LOI of polypropylene using an intumescent system comprising only two components (aldehyde resin + MP – Ex 1) and (aldehyde resin + APP – Examples 4 & 5).

(ii) It is also possible to improve the LOI of polypropylene with three component systems (Examples 2 and 3).

(iii) The most successful results are with four component intumescent systems (Examples 6, 7, 8 and 9).

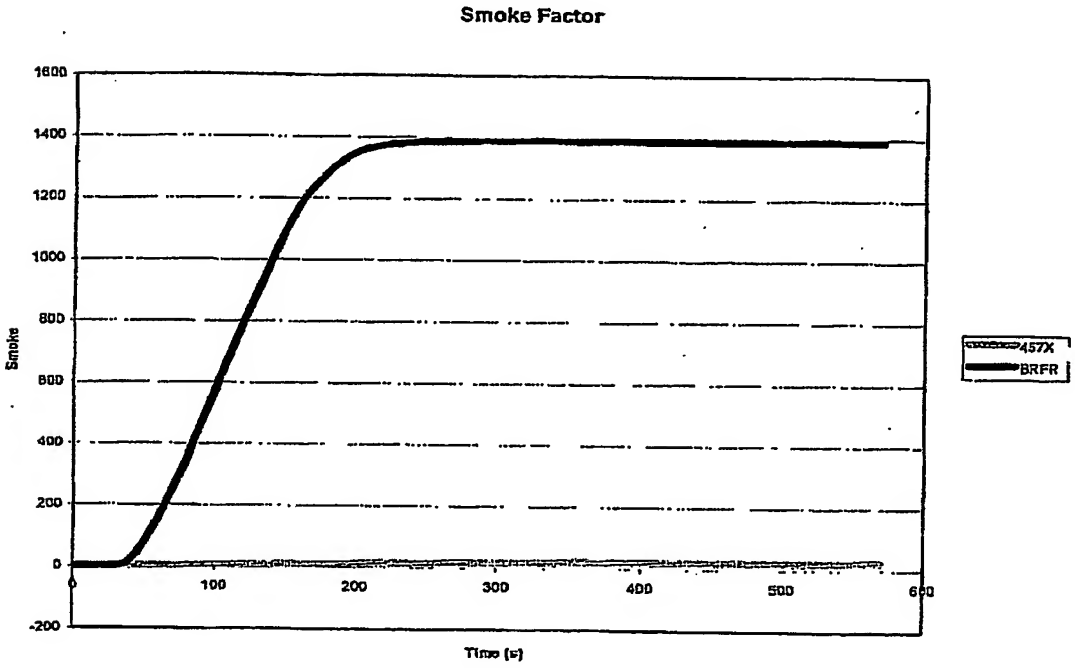
(iv) Table 2 shows that formulations can be prepared to achieve LOI results of 33.2.

(v) Table 3 shows that UL94 VO ratings can be achieved at a thickness of 0.8mm.

There now follows graphs depicting the smoke reduction, heat release and MFI properties of the flame retardant product of the invention. In the graphs, the flame retardant product designated 457X is the four component sample of Example 9 in Table 2.

GRAPH A

Smoke Factor

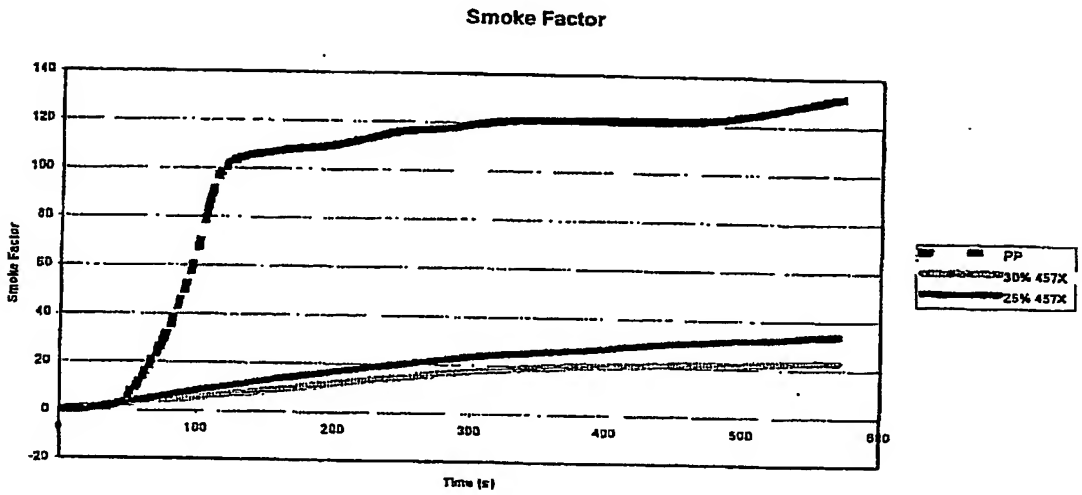


Cone Calorimeter Cumulative Smoke Results (50kW)

Flame retardant of Example 9 incorporated at 30% in polypropylene. BRFR is a polypropylene flame retarded with 21% decabromo diphenyl ether and 12% antimony trioxide.

GRAPH B

Smoke Factor



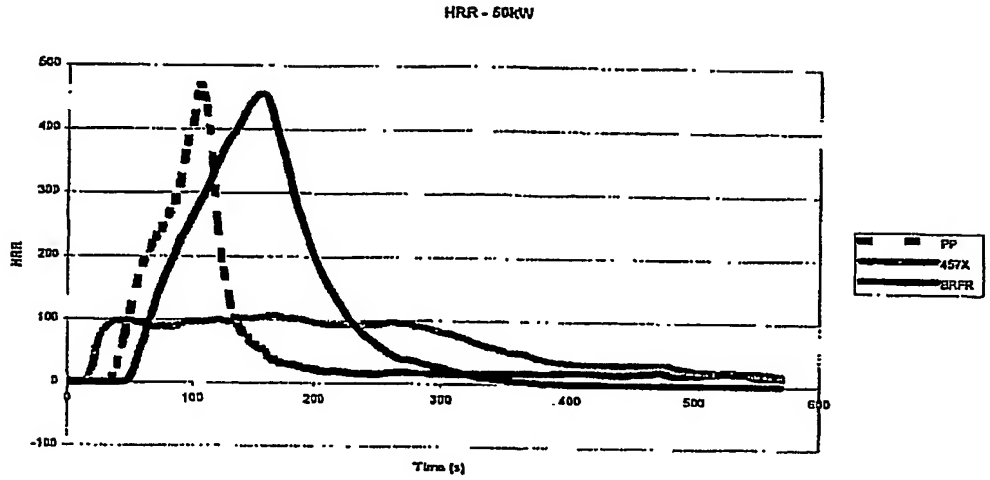
Cone Calorimeter Cumulative Smoke Results (50kW)

PP=Polypropylene, flame retardant of Example 9 included at 25% and 30% in PP

The Graphs A and B above show the significant reduction in cumulative smoke, which can be achieved when the flame retardant product of the invention is incorporated in PP. The smoke produced from the polymer can be reduced by up to 75%. More significantly however, when compared to brominated flame retardants systems, the invention produces only 2% of the total smoke evolved from the brominated systems. This is an extremely important factor as while a fire is developing, smoke is as big a threat to life as the fire is itself.

GRAPH C

HRR – 50kw

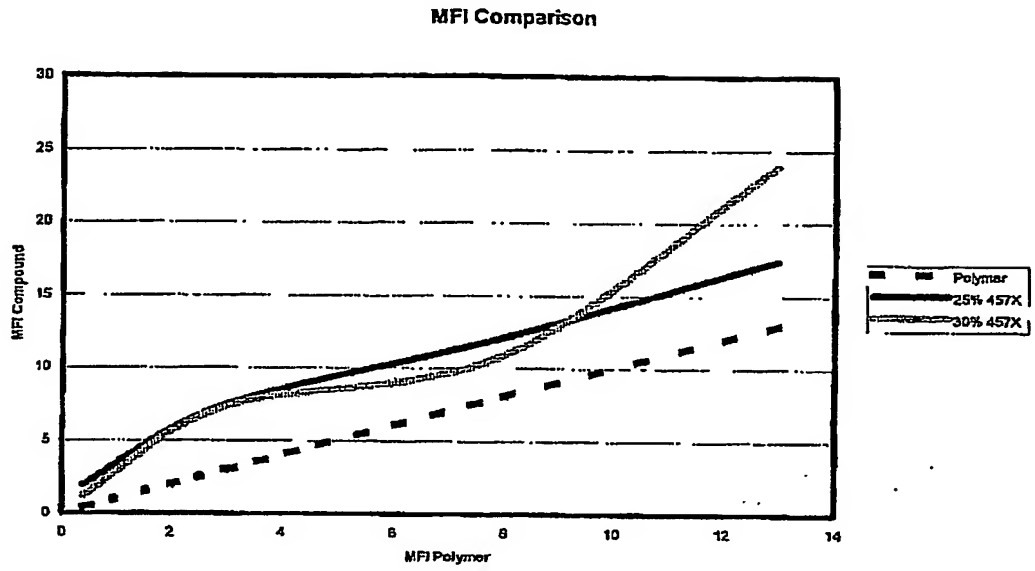


Cone Calorimeter Heat Release Rates (50kW)
PP=Polypropylene. Flame retardant of Example 9 incorporated at 30% in polypropylene. BRFR is a polypropylene flame retarded with 21% decabromo diphenyl ether and 12% antimony trioxide.

Graph C shows the heat release rates for three systems, untreated PP, PP treated with brominated flame retardants and PP treated with the flame retardant invention. The heat release rate is important as it shows how much energy a system will add to the development of a fire. The untreated PP shows a significant peak after 100s, which demonstrates it will contribute significantly to any fire development in the early stages. The brominated flame retardant works by increasing the resistance to ignition, which is demonstrated in the graph by the delay in reaching the peak HRR by 60 seconds. However, once ignited this system liberates as much energy as the untreated PP at the maximum HRR peak. The system containing the flame retardant of the invention shows no significant peak in the HRR and plateaus at a level 70% below the peak HRRs of the untreated PP and brominated flame retardant systems. This demonstrates that the flame retardant of the invention when incorporated in a polymer will not only help increase the resistance to ignitability but will also reduce the contribution the polymer will make to the development of a fire.

GRAPH D

MFI Comparison



Melt Flow Index (MFI) Comparisons (230°C, 2.19kg)
Polymer=polypropylene, flame retardant of Example 9 included at 25% and 30% in PP.

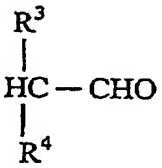
Melt Flow Index measures the amount of polymer which can be extruded in a 10 minute period, for a given temperature and force/weight. Essentially this can be considered as a measure of how easily a given polymer can be processed, the higher the value the easier processing should be. Graph D shows the effect of adding the flame retardant of the invention to PP. In general the addition of the flame retardant of the invention will increase the MFI, so making it easier to process the polymer.

CLAIMS

1. A flame retardant composition which comprises a mixture of a phosphorus containing compound which decomposes to produce phosphoric acid when exposed to flame and an oxygenated thermoplastic heterocyclic resin which is prepared by reacting an urea of the general formula (I)

(I)

where X is oxygen or sulphur and R^1 and R^2 are hydrogen, identical or different alkyl of 1 to 18 carbon atoms, aryl of 6 to 9 carbon atoms or aralkyl of 7 to 9 carbon atoms or may be an alkyleneurea radical, where alkylene is of 1 to 9 carbon atoms, with at least 2 moles of a CH-acidic aldehyde of the formula (II)



(II)

where R^3 is hydrogen and R^4 is alkyl, aryl or aralkyl, or R^3 and R^4 are identical or different alkyl, aryl or aralkyl, in the presence of a strong acid, to give a condensation product, and thereafter treating the product with an alkali metal alcoholate in an anhydrous medium.

2. A flame retardant composition as claimed in claim 1 wherein the compound of general formula (I) is urea.

3. A flame retardant composition as claimed in claim 1 or claim 2 wherein R3 and R4 in the compound of general formula (II) is alkyl of 1 to 10 carbon atoms, aryl of 6 to 9 carbon atoms or aralkyl of 7 to 9 carbon atoms.

4. A flame retardant composition as claimed in any one of claims 1 to 3 wherein the compound of general formula (II) is isobutyroaldehyde.

5. A flame retardant composition as claimed in any one of the preceding claims wherein the phosphorus containing material is selected from ammonium polyphosphate, sodium polyphosphate, potassium polyphosphate, melamine polyphosphate or mixtures thereof.

6. A flame retardant composition as claimed in claim 5 wherein the phosphorus containing material is a mixture of ammonium polyphosphate and melamine phosphate.

7. A flame retardant composition as claimed in any one of the preceding claims which contains a blowing agent which produces a non-flammable gas when exposed to flame.

8. A flame retardant composition as claimed in claim 7 wherein the blowing agent is melamine or urea.

9. A flame retardant composition as claimed in any one of the preceding claims wherein the phosphorus containing material is encapsulated in the oxygenated heterocyclic thermoplastic resin.

10. A flame retardant composition as claimed in any one of claims 7 to 9 wherein the blowing agent also is encapsulated in the oxytenated heterocyclic thermoplastic resin.

11. A flame retardant composition as claimed in any one of the preceding claims which is prepared by extrusion

12. A flame retardant composition as claimed in any one of the preceding claims wherein the flame retardant composition comprises 25 to 60% by weight oxygenated heterocyclic thermoplastic resin; 0 to 75% by weight ammonium polyphosphate; 0 to 75% by weight melamine phosphate and 0 to 45% by

weight melamine with the proviso that ammonium polyphosphate or melamine phosphate essentially is present.

13. A composition of matter which contains a flame retardant composition as claimed in any one of claims 1 to 12.

14. A composition of matter as claimed in claim 13 wherein the flame retardant composition is present in the composition of matter in an amount of 5 to 90% by weight.

15. A composition of matter as claimed in claim 14 where the flame retardant composition is present in the composition of matter in an amount of 10 to 45% by weight.

16. A composition of matter as claimed in any one of claims 13 to 15 selected from thermoplastic polymers, thermosetting polymers, paper, reconstituted wood products and solvented systems.

17. An composition of matter as claimed in claim 16 which is a polyolefin.

18. An composition of matter as claimed in claim 17 wherein the polyolefin is polypropylene.

20. An article formed from a composition of matter as claimed in any one of claims 13 to 18.

21. An article as claimed in claim 20 formed by injection moulding or compression moulding.

22. A method of improving the flame retardant capability of a composition of matter by embodying in the composition of matter a flame retardant composition as claimed in any one of claims 1 to 12.